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EXTRUSION OF IRON ORE USING POLYMERS AND CALCIUM CARBONATE AS PLASTICIZER

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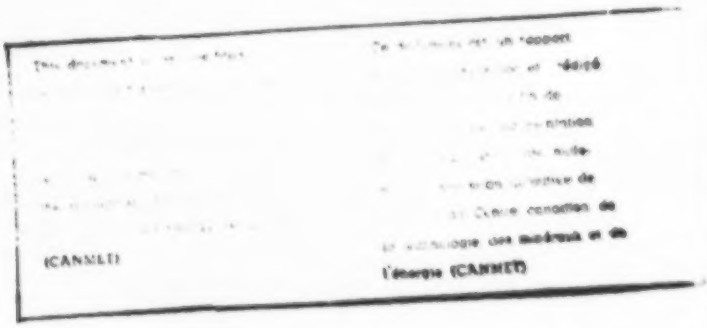

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EXTRUSION OF IRON ORE USING POLYMERS AND CALCIUM CARBONATE AS PLASTICIZER

by

D.H.H. Quon* and K.E. Bell**

ABSTRACT

Pellets were fabricated from the Mt. Wright iron ore concentrates by vacuum extrusion using polymers and calcium carbonate as plasticizer and binder. Favourable green and dry crushing strengths of the pellets were obtained. At low and middle induration temperature intervals the crushing strength is unfavourable due to the decomposition of the polymer. At high induration temperature, the strength of the pellets containing polymer and calcium carbonate surpasses those containing bentonite.

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EXTRUSION DU MINERAI DE FER À L'AIDE DE POLYMÈRES ET DE CARBONATE DE CALCIUM COMME PLASTIFIANTS

par

D.H.H. Quon* et K.E. Bell**

RÉSUMÉ

On a fabriqué des boulettes à partir de concentrés de minerai de fer provenant du Mont Wright par extrusion sous vide en utilisant des polymères et du carbonate de calcium comme plastifiants et liants. L'écrasement grain par grain des boulettes sèches ou humides se réalise bien. À des intervalles de température de durcissement bas ou moyen, l'écrasement grain par grain est difficile en raison de la décomposition du polymère. À une température de durcissement élevée, la résistance des boulettes contenant des polymères et du carbonate de calcium est supérieure à celle des boulettes contenant de la bentonite.

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INTRODUCTION

The role of bentonite addition for agglomeration and extrusion of iron ore is to give the pellets dry, preheated and high-temperature strength to ensure their survival in handling. However, the addition of this material has a detrimental effect as it tends to decrease the iron content of the pellet and contributes to acid gangue in the finished product. The presence of excess acid gangue from the bentonite requires additional quantities of fluxes and also affects the reducibility during the ironmaking process. In addition, the supply of high quality bentonite is being depleted; the use of lower quality material poses a requirement for increased amounts of bentonite addition (with increased impurity levels) as well as contributing to greater variation in the physical properties of the pellets. In order to negate these problems, alternative high-temperature binders to replace bentonite were examined. In an earlier report, it has been demonstrated that iron ore pellets can be successfully extruded using bentonite and polymers as plasticizer and binder (1). The aim of this work is to substitute other binders for bentonite in iron ore extrusion. Another advantage of the extrusion process is that the iron oxide can be shaped to produce pellets with uniform physical characteristics. This paper will focus on the use of organic and inorganic binders for iron ore extrusion.

GENERAL CHARACTERISTICS OF BINDERS

Bentonite is considered as one of the best low- and high-temperature binders for pelletizing of iron ore. It provides sufficient green and fired strength to the pellets. In the extrusion process, bentonite also serves as a plasticizer to aid in the extrusion. There are very few inorganic binders having physical properties similar to bentonite; however a variety of inorganic binders have been used as plasticizers and to provide green strength for clay-free materials, enabling them to be molded into desirable shapes before firing. These binders have been used extensively in ceramic processes, including dry pressing, extrusion, tape casting, injection molding and compression molding. A description of the

various binders has been documented in the literature (9-14). They were classified based on their origin, chemical composition, physical characteristics, molecular weight, polarity, affinity for water, and dominant adhesion mechanisms.

Consideration of the suitability of the various available binders that may be used for iron ore extrusion indicates that the binder must be readily soluble or dispersable in a liquid (either organic or water) and free from contamination of the iron ore. A uniform dispersion of the liquid phase is important for distributing the binder throughout the particulates and to provide fluidity for slips or plasticity for extrusion. Dry green strength in the product is developed by the evaporation of the liquid. The binder is retained in the product and provides organic bridges between particles.

The binders can strongly affect the rheology of the liquid phase. They increase the viscosity and change the flow characteristics from Newtonian to pseudoplastic in most cases. Also, with some binders, gels can develop under various conditions. The rheology of the solution directly affects the behaviour of suspensions and pastes formed by adding particulates to the solution. It is readily apparent that the applicability of a given binder for a specific process is very much dependent on the rheological characteristics of the binder solution.

EXPERIMENTAL PROCEDURE

Raw Materials

The iron oxide used for the experimental work was obtained from Mt. Wright, Quebec. The concentrates were composed of essentially specular hematite with a trace of magnetite, feldspar and quartz.

High-temperature binders alternative to bentonite that were used for the experimental work included calcium oxide, calcium carbonate, and calcium hydroxide. The amount of these additive used is normally less than 3 mass%.

Organic binders used as plasticizers and low-temperature binders in this study were: carboxymethylcellulose (CMC), methylcellulose (MC), hydroxyethylcellulose (HEC), sodium and ammonium alginates. Table 1 gives a list of the source and description of the organic binders used for the experimental work.

The plasticity of the iron ore with binder or binder systems was determined by using a Barbender Plastograph similar to the study described previously (1). Factors affecting plasticity that are considered include amounts of binder, binder combination, and the water content to produce optimum consistency for extrusion. The plasticity of the material was measured in terms of consistency, and this value was obtained from the Plastograph. For successful extrusion, the minimum consistency value is about 450 m.g of torque. The term consistency is a measure of the plastic viscosity of the flowing plastic dispersion.

Only mixtures containing binders and iron oxide with a consistency value about 450 m.g of torque were used for the extrusion experiments. Appropriate amounts of the constituents were weighed and mixed with the required amount of water in a blender until a uniform dispersion of the individual components in the mix was obtained.

Extrusion work was conducted in two types of vacuum extruders. Preliminary extrusion work was conducted in a piston type extruder, where the amount of mixture used is less than 500 g (Fig. 1). An auger type extruder was used to extrude batch sizes above 4 kg (Fig. 2). Extrusion was conducted in a vacuum to remove the occluded air and maintained at about 63 cm of Hg. The die used for the piston type extruder was square, having dimensions of 2.5 x 2.5 cm. For the auger extruder, the die was made up of a three-hole taper-nose having hole diameters of about 2.54 cm. A piano-wire cutting-off machine was used to cut the desirable cylindrical pellet length after the mixture was extruded from the die.

The pellets formed by extrusion were dried in an oven for 24 h at 120°C. On completion of the drying cycle, the crushing strength of the pellets was determined. Some of the oven dried pellets were then indurated in a resistance furnace at temperatures from 950 to 1300°C for periods of 10 and 20 minutes. The rate of heating of the pellets from room temperature to the induration temperature was normally less than 10 minutes. On completion of the induration cycle, the pellets were removed from the furnace and covered by mineral fibres to reduce the thermal shock due to a rapid rate of cooling. Following cooling, the strength of the pellet was determined.

The reaction of the binders and iron ore at various temperature was determined using the differential thermal analysis technique (DTA).

Microstructure of the indurated pellets was examined using an optical microscope.

RESULTS

Plasticity of the Organic and Inorganic Binders

The peak consistency of the Mt. Wright iron ore alone as measured by the Plastograph is only 290 m.g of torque (Fig. 3). With MC being added to the iron ore, this value continued to increase, reaching a consistency value of 1600 m.g of torque at an additive concentration of 1.5% (Fig. 3). The consistency of other binders added to the iron ore concentrates is given in Table 2.

With the addition of 0.25 mass% MC to the iron ore, it developed a consistency value of 750 m.g of torque. At this consistency level, it is possible to extrude the iron ore. One of the major problems is that the low decomposition temperature of the organic binders may affect the low-temperature strength of the pellets. In order to develop adequate pellet strength to withstand the load during induration, an additional binder might be required due to the loss of the organic binder. It is also assumed that at the high temperature sufficient solid state reaction takes place, such as recrystallization and grain growth that comprise the major bonding mechanisms, to produce strong pellets. It appears that a moderate- to high- temperature binder (400 to 1100°C) is required to bond the particulates after the organic binder burns off. The criteria in the selection of a suitable high temperature binder to replace bentonite require that it forms a bond with iron oxide at a moderate induration temperature without the formation of a liquid phase resulting from the reaction of the binder with the ore, and that it develops sufficient porosity to allow the gas to permeate the pellet during the subsequent reduction process. Compounds such as calcium oxide, calcium hydroxide and calcium carbonate appear to be suitable as they react with the iron oxide to form calcium ferrite at a moderate temperature (1180°C) and this phase can also act as a bonding agent for the iron oxide grains (2).

The consistency of iron ore with variable amounts of calcium compounds and organic binders was first investigated. Table 2 gives the consistencies achieved with various binder systems of this type. The results revealed that the consistency of the iron ore is dependent on the quantity of the organic binder and the type of calcium compound added.

The consistency versus water added to the iron ore with 0.25 mass % MC and 1 to 2% CaO, $Ca(OH)_2$ and $CaCO_3$ is graphically shown in Fig. 4 and 5. A common characteristic of these curves is a high sharp rising peak at a level of 10-12% water addition. In general, a higher consistency value was obtained from mixtures containing $CaCO_3$ with an organic binder than those containing CaO or $Ca(OH)_2$ with an organic binder (Fig. 5). A mixture containing 0.25 mass% MC with $CaCO_3$ gave a peak consistency value of above 900 m.g of torque. This value decreased with reduction in the quantity of organic binder. The consistencies obtained by adding either HEC or alginate along with $CaCO_3$ are plotted in Fig. 6, which indicates that HEC is the more effective plasticizer in this system. For example, 0.5% alginate is required to produce a consistency of about the same magnitude as with 0.25% HEC (Fig. 6). The difference in the plasticity development appears to relate to the viscosity difference of the two organic binders. The effect of different grades of CMC on the consistency of the iron ore containing $CaCO_3$ was examined. The results are plotted in Fig. 7 and indicate that the low viscosity grade CMC produced a lower consistency, compared with CMC of a higher viscosity grade.

As previously reported, a minimum consistency value of 450 m.g of torque is required for iron ore extrusion, and it appears that $CaCO_3$ in combination with certain organic binders can be used effectively as a plasticizer/binder system.

Strength development of pellets

Of the different calcium compounds examined as a possible replacement for bentonite, calcium carbonate was chosen as it improves the consistency of the iron ore containing small quantities of organic binder. Extrusion characteristics for iron ore containing MC, CMC, HEC and alginate and 1 to 2 mass% $CaCO_3$ are summarized in Table 3. The results revealed that the green pellets containing HEC or CMC tend to deform in the drop test or under their own weight due to the excess water required. The dry strength

of the pellets shown in Table 4 indicates that pellets containing alginate and HEC are stronger than those containing MC and CMC.

Prior to the induration of the extrudates, DTA was used to examine the reaction of the various binders and iron oxide. The resulting curves, shown in Fig. 8, indicate that the organic binder decomposes at a temperature below 400°C as revealed by the exothermic peak at about 350°C. The endothermic peaks at 576, 680 and 800°C represent the transformation of quartz, Curie temperature of the iron oxide, and decomposition of the $CaCO_3$ respectively.

The strength development of the pellets containing $CaCO_3$, MC, CMC, HEC and alginate indurated at different temperature and time are given in Table 4. It can be seen that at an induration temperature of 1050°C, the strength is considerably lower than the dry pellet strength. This is to be expected, as the higher dry strength is developed due to binding by the polymer phase which holds the iron oxide particulates together. With increasing induration temperature, the organic binder decomposes first and the bonding of the particulates becomes weaker. Based on the strength development of the pellets, the decomposition of $CaCO_3$ to CaO takes place below 900°C but the CaO does not react with the iron oxide until above 1100°C. It appears that at low induration temperature solid state reaction between these two components is minimum. The higher strength developed at high induration temperature appears due to the minor liquid phase bonding resulting from the reaction of iron oxide, CaO and the free silica in the iron ore concentrates.

The pellet strength development with temperature and time, as shown in Fig. 9 and 10, indicates that both MC and HEC developed higher strength than alginate and CMC. Furthermore, extrudates containing alginate always have a lower pellet strength than extrudates containing other binders under identical heat treatment. The minimum induration temperature and time for adequate pellet strength (2000N) is 1175°C for 20 min or 1300°C for 10 min.

Two types of CMC were used as plasticizers, the difference being viscosity. The strength of the pellets using high viscosity CMC is higher than for low viscosity CMC under identical heat treatments.

The development of pellet strength for the several binder systems was traced from low to high induration temperature; the results obtained are

plotted in Fig. 11. For comparison, using bentonite alone (or as a component of mixed binder systems) the strength increases continuously with temperature. On the other hand, using organic binders and $CaCO_3$, strengths below the dry strengths were found at about 500°C. The reduction of strength between 120°C and 500°C is due to the loss of organic binder. This was followed by a rapid increase in strength after being fired above 800°C. It appears that, at this low induration temperature, commencement of bridging of iron oxide grains occurred. Of the four organic binders used, the weakest low temperature strength was observed in the mixture containing MC and the highest for alginate and CMC.

Microstructural examination of the indurated pellets showed that, under identical heat treatment, the large specular hematite grains are modified from a sharp angular to a rounded shape due to reaction, in a high-temperature liquid phase, between iron oxide, CaO and free silica (Fig. 12 to 14). Extensive microstructural modification of the matrix phase can be seen in the micrographs. Here, the fine-grained iron oxides, occurring mainly as interstitial material between the platelets of specular hematite, are also bonded by this liquid phase.

DISCUSSION

The potential use of organic binders for iron ore balling has been reported by other investigators (6-11). Satisfactory pellets have been produced using a cellulose based polymer (Peridur XC-3*) at levels ranging from 0.1 to 0.4 mass% substituted for bentonite. Pilot plant tests conducted in Cia Vale Do Rio Doce (CVRD) using Peridur binder produced fired pellets of acceptable physical characteristics as specified by the ISO standard (12).

In the present work, the major aim has been to extrude iron ore pellets, which is different from the balling process. Higher density pellets can be obtained as a result of pressure forming, as compared with the balling technique. Research literature on the extrusion of ceramic powders is quite limited, and most studies involved compositions containing clay. When clay-free material is used, rheological properties similar to those of clay bodies may be developed with organic or inorganic additives.

The mechanism involved in the process is formation of a gel-like substance on the surface of the iron oxide from the reaction of water and polymer. The gel provides lubrication so that a dispersion of solid particles may exhibit flow characteristics similar to the non-newtonian fluids; i.e., the system has a fluid phase that has sufficient viscosity to prevent the individual particles from directly contacting each other. The binders used for this investigation may be classified into:

a) soluble additives, and
b) non-soluble additives

Cellulose based polymers and alginates are good plasticizers, and the extrudates developed good green and dry strength. The degree of plasticity imparted by these polymers depends on their viscosity. They also performed satisfactorily as low temperature binders. Insoluble calcium compounds are not considered to contribute directly to plasticity, but they tend to modify the consistency of iron ore containing organic binders, as demonstrated with CaO and $Ca(OH)_2$ additions. The $CaCO_3$ is a high temperature binder as it improves the strength of pellets indurated at high temperatures.

The ability of the polymers to develop proper plasticity for pellet extrusion is a result of a monomolecular film of the binder phase covering the surfaces of the iron oxides. The extrusion efficiency becomes maximum with an increase in either binder concentration or viscosity. The polymers are composed of long chain molecules, which can be varied as regards number and spacing; the viscosity of these polymers can also be varied dependent on the solution concentration. The increase in viscosity of a liquid as large molecules (and colloids) are added is strongly influenced by their shape. The effectiveness of such molecules or colloids is dependent on their "sphere of influence" in the liquid.

Both cellulose based polymers and alginates are found in medium- to high-viscosity ranges. The viscosity, η, of the solution is related to the concentration, c, of the binder as follows (13):

$$\log \eta = kc$$

where η is expressed in centipoise, c is weight per cent, and k is a measure of viscosity grade.

For good extrusion of pellets, it is necessary to use polymers of high viscosity grade and the viscosity of the liquid present must be high. If the liquid is too fluid, it would be easily squeezed out and separated from the iron ore particulates during the extrusion process, giving rise to high friction and poor flow.

Experiments conducted with polymers and $CaCO_3$ binder have demonstrated that the iron ore concentrates from Mt. Wright can be extruded successfully. Although unfavourable crushing strengths were obtained for pellets fired at the lower and middle temperature intervals, due to the decomposition of the organic binder, the strength of pellets containing $CaCO_3$ and indurated at high temperatures surpasses the strength of all other pellets including those containing bentonite.

CONCLUSION

Specular hematite from Mt. Wright, when mixed with a polymer in solution, behaves as a plastic dispersion mass much the same as clay and water. That is, the system has a liquid phase that lubricates the iron oxide particles and that has sufficient viscosity to prevent the individual particles from contact.

It has also been demonstrated that extrusion of pellets may be achieved by using polymers in combination with $CaCO_3$. The polymers also provide good dry strength for the pellets. However, on the decomposition of the polymer, the pellet strength decreases, resulting in an appreciable lowering of the strength. The strength appears to recover above 800 °C and increases rapidly with further increase in temperature. Pellets containing polymers and $CaCO_3$ tend to develop higher strength at high temperature of induration than pellets containing bentonite and bentonite with polymer additions.

ACKNOWLEDGEMENT

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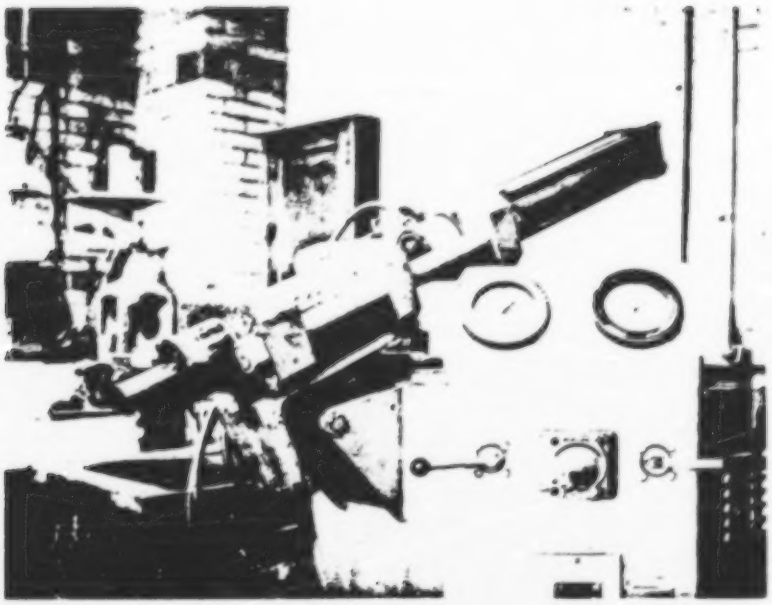

Fig. 1. Extruder used for small-scale pellet extrusion

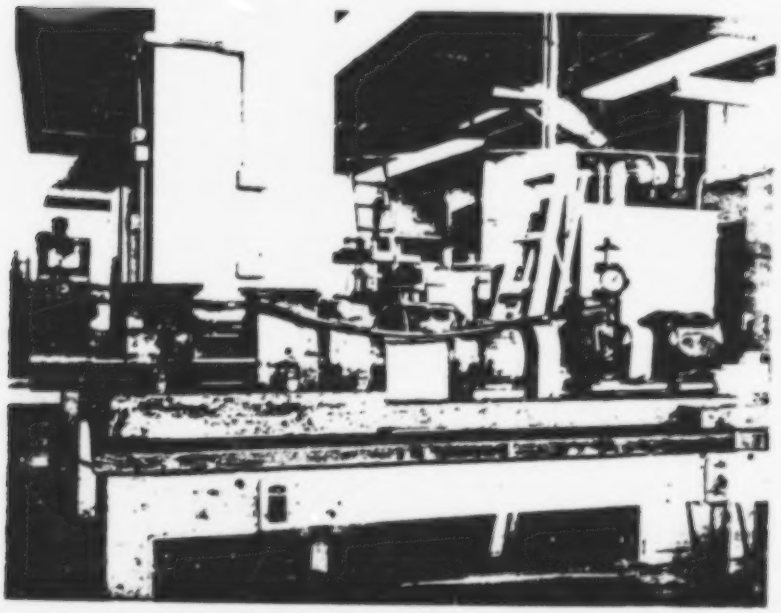

Fig. 2. Extruder used for large-scale pellet extrusion

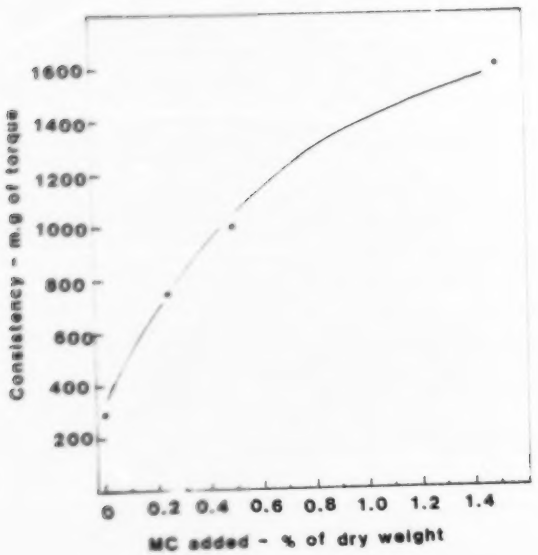

Fig. 3. Effect of MC concentration on the consistency of iron ore

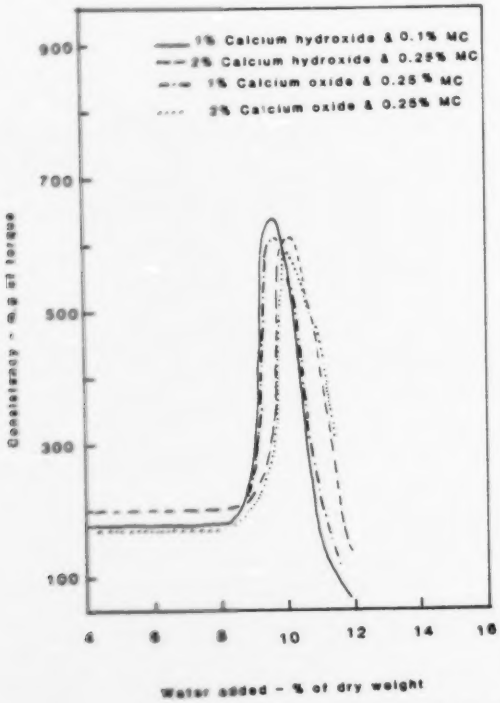

Fig. 4. Effect of calcium oxide, calcium hydroxide and MC addition to iron ore

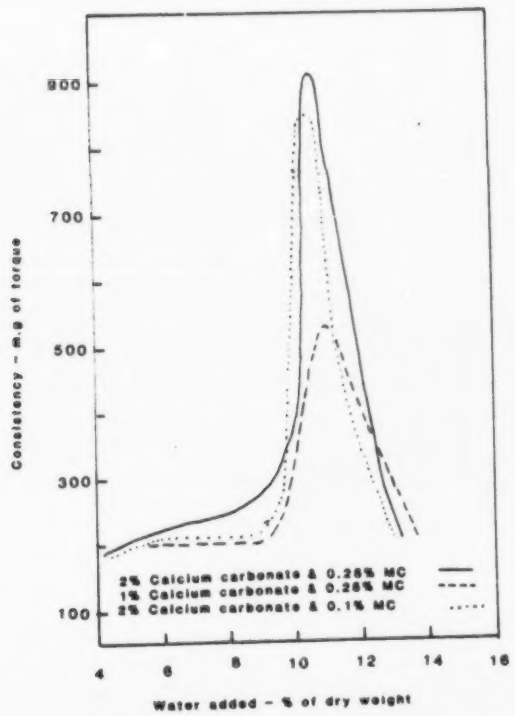

Fig. 5. Effect of calcium carbonate and MC additions to iron ore

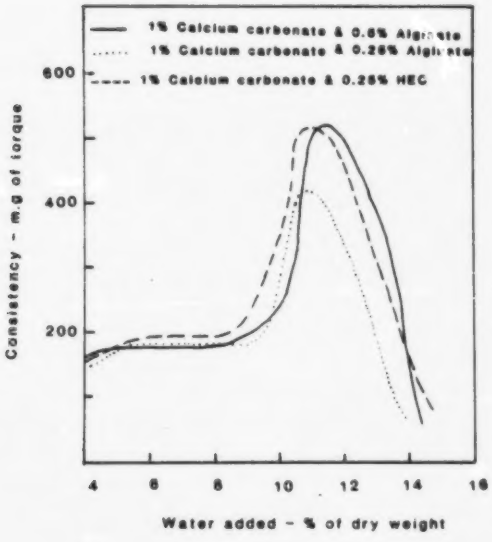

Fig. 6. Effect of calcium carbonate, HEC and alginate additions to iron ore

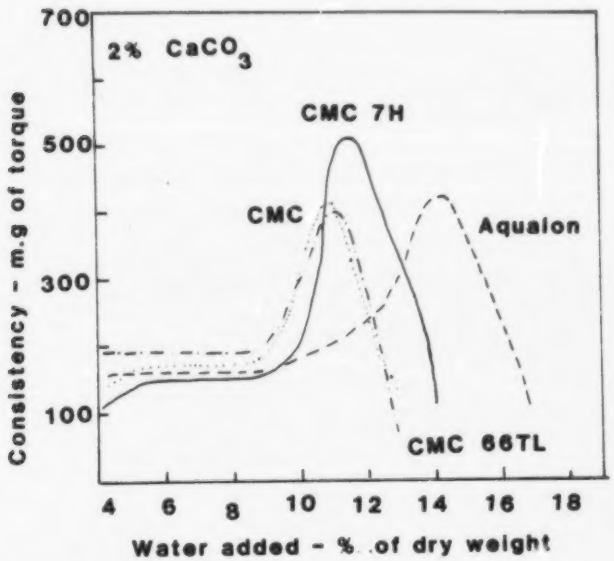

Fig. 7. Effect of calcium carbonate and various grade of CMC additions to iron ore

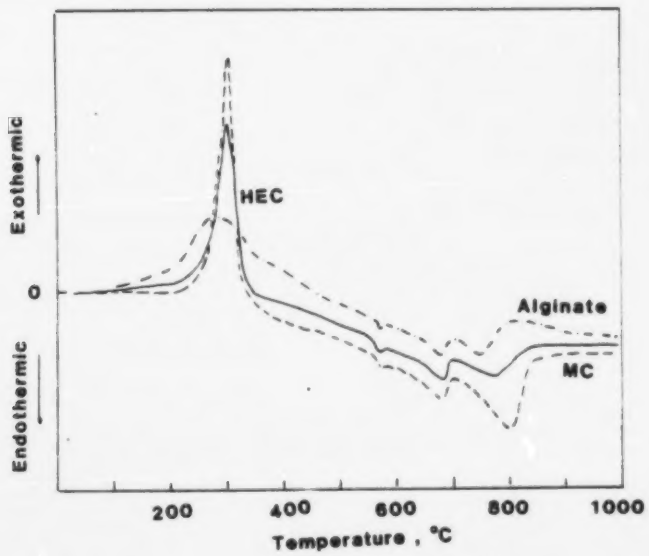

Fig. 8. DTA curves for iron ore containing different binders

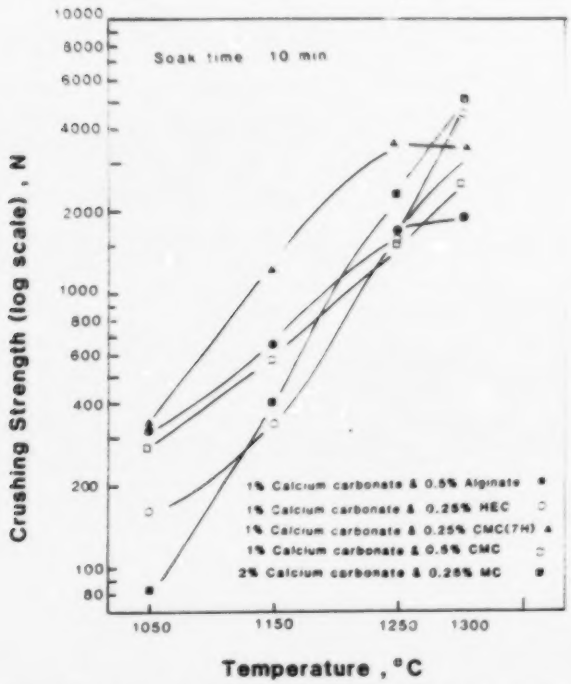
Soak time 10 min
Crushing Strength (log scale) , N
Temperature , °C
1% Calcium carbonate & 0.5% Alginate
1% Calcium carbonate & 0.25% HEC
1% Calcium carbonate & 0.25% CMC(7H)
1% Calcium carbonate & 0.5% CMC
2% Calcium carbonate & 0.25% MC

Fig. 9. Strength development of pellets indurated at various temperatures for 10 min.

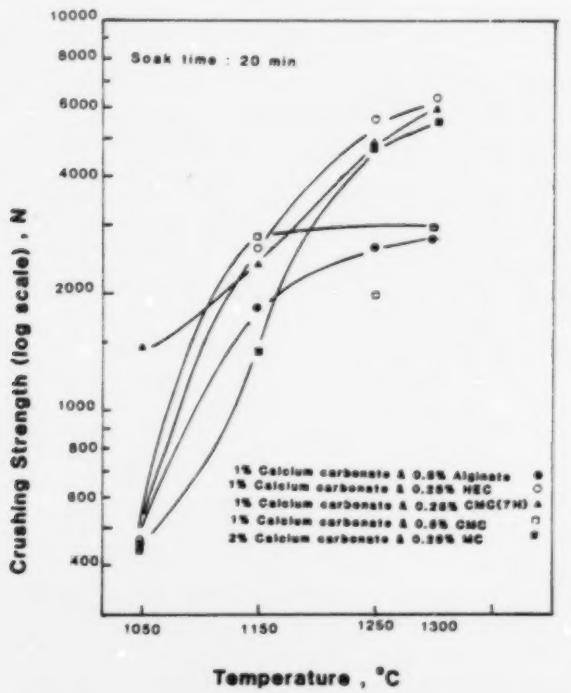
Soak time : 20 min
Crushing Strength (log scale) , N
Temperature , °C
1% Calcium carbonate & 0.5% Alginate
1% Calcium carbonate & 0.25% HEC
1% Calcium carbonate & 0.25% CMC(7H)
1% Calcium carbonate & 0.5% CMC
2% Calcium carbonate & 0.25% MC

Fig. 10. Strength development of pellets indurated at different temperatures for 20 min.

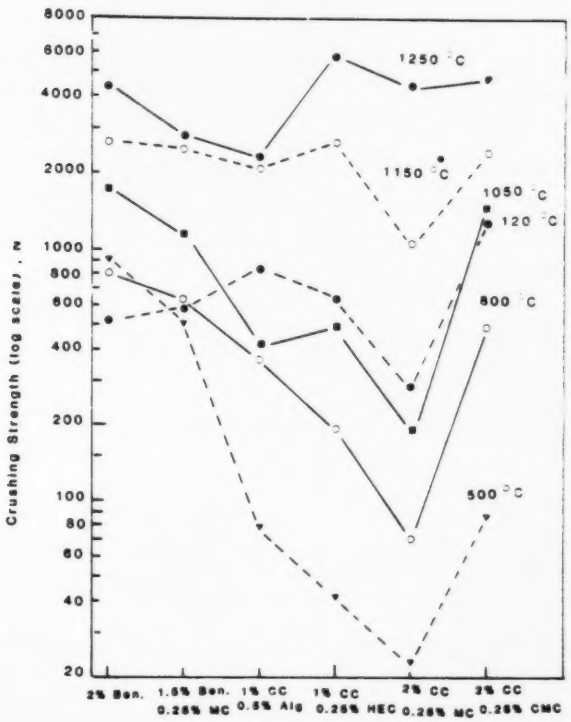

Fig. 11. Strength of pellets with various binder additions as a function of the induration temperature for 20 min.

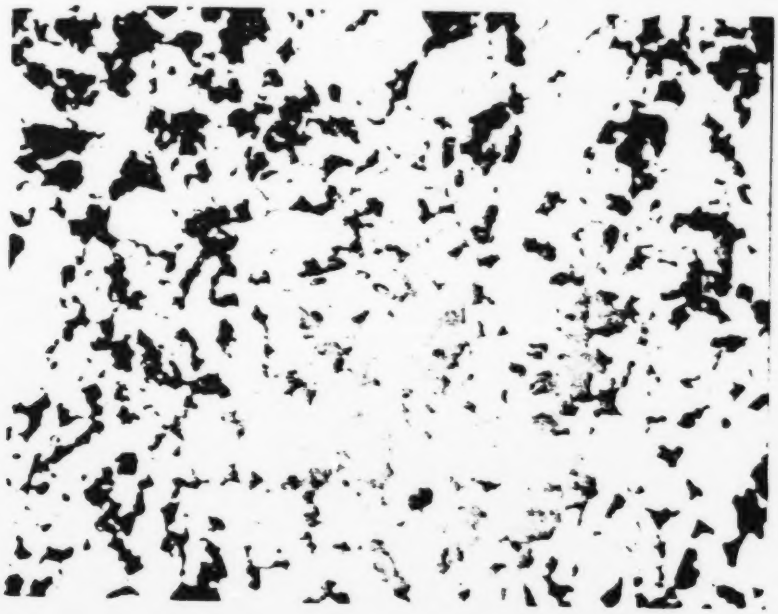

Fig. 12. Microstructure of pellet containing MC and calcium carbonate indurated at 1300°C for 20 min.

Fig. 13. Microstructure of pellet containing HEC and calcium carbonate indurated at 1300°C for 20 min.

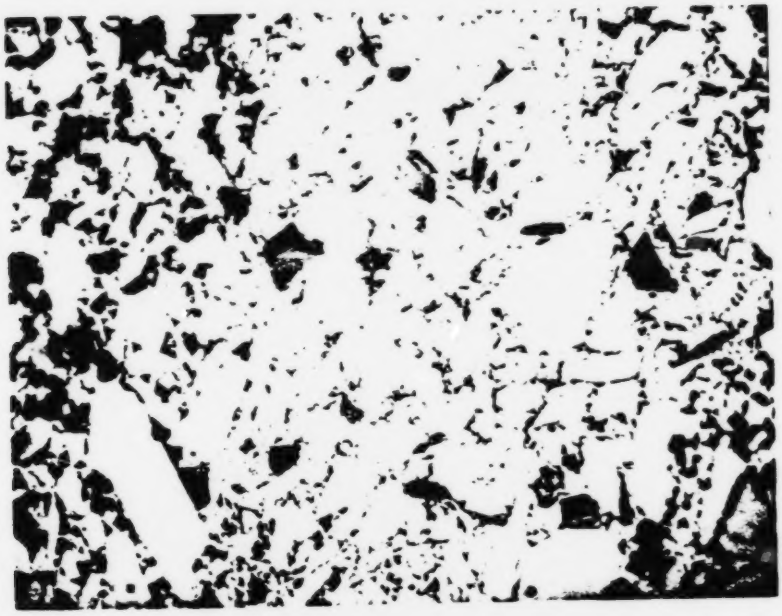

Fig. 14. Microstructure of pellet containing alginate and calcium carbonate indurated at 1300°C for 20 min.

Table 1. Source and description of products

Product	Chemical Identity	Manufacturer
Aqualon	Carboxyl methycellulose	Hercules
Acrysol A-3	Acrylic	Rohm & Haas
Acrysol G110	Acrylic	Rohm & Haas
Acrysol AC-33	Acrylic	Rohm & Haas
Alginate (Na)		Kelco
Calcium hydroxide		Fisher Chemical
Calcium carbonate		Fisher Chemical
Calcium oxide		Fisher Chemical
Carboxyl methylcellulose (Na)		Hercules
CMC-7H	Carboxyl methylcellulose	Hercules
CMC 6CTL	Carboxyl methylcellulose	Hercules
Methocel	Methylcellulose (3000 cps)	Dow Chemical
Methocel F4M	Hydroxypropyl methylcellulose	Dow Chemical
Miniflo	Guar Gum	Hercules
Natrosol	Hydroxyl ethylcellulose	Hercules
Polysar PL404	Latex	Polysar Co.
Polysar PL458	Latex	Polysar Co.
Polysar PL776	Latex	Polysar Co.
Polysar PL989	Latex	Polysar Co.
Superloid	Amomonium alginate	Kelco Co.

Table 2. Determination of plasticity for iron ore extrusion

Inorganic Binder, %		Organic Binder, %	Water, %	Plasticity,+ peak height
1.0 cc	1.0	p1404 polysarlatex	11.5	400
1.0 cc	0.5	p1404 polysarlatex	11.5	360
1.0 cc	1.0	p1989 polysarlatex	11.0	350
1.0 cc	0.5	p1989 polysarlatex	11.5	320
1.0 cc	2.0	p1404 polysarlatex	11.0	390
1.0 cc	2.0	p1989 polysarlatex	12.0	390
1.0 cc	3.0	p1458 polysarlatex	6.5	310
1.0 cc	2.0	p1776 polysarlatex	8.0	320
1.0 cc	1.0	p404 polysarlatex & 1.0 tween 80	9.0	270
1.0 cah	0.25	methylcellulose	9.4	640
2.0 cah	0.25	methylcellulose	9.8	600
1.0 cah	0.25	methylcellulose	10.0	610
2.0 cao	0.25	methylcellulose	10.5	580
2.0 cc	0.25	methocel F4M	10.5	720
1.0 cc	0.25	methylcellulose	10.8	850
2.0 cc	0.25	methylcellulose	11.9	910
1.0 cc	0.5	alginate	11.5	510
1.0 cc	0.25	HEC	11.0	510
1.0 cc	0.25	CMC	11.0	395
2.0 cc	0.25	CMC 6CTL	11.0	400
2.0 cc	0.25	Aqualon	14.5	420
2.0 cc	0.25	CMC-7H	11.5	505
1.0 cc	1.0	acrysol G110	11.0	390
1.0 cc	0.25	alginate	8.1	410
2.0 cc	0.25	maniflo	11.0	375

*inorganic binder:
cah = calcium hydroxide, cc = calcium carbonate, cao = calcium oxide

+consistency in meter-grams or torque

Table 3. Characteristics of green pellets

Extrusion No.	Binders and Water	Characteristics
1	1% $CaCO_3$ & 0.5% alginate 11% water	Moderate wet strength
2	1% $CaCO_3$ & 0.5% alginate 10% water	Good wet strength, no deformation
3	1% $CaCO_3$ & 0.25% HEC 10.5% water	Deformed pellets
4	2% $CaCO_3$ & 0.25% MC 10% water No vacuum	Deformed pellets
5	2% $CaCO_3$ & 0.25% MC 10% water	Pellets not deformed
6	1% $CaCO_3$ & 0.5% CMC 12% water	Pellets not deformed
7	2% $CaCO_3$ & 0.25% CMC-7H 12% water	Pellets deformed

*deformed refers to pellet changes in shape on dropping

Table 4. Crushing strength of the indurated pellets

Ext. No.	Binders	Dry strength+, N	Indurated temp, °C	time, m	Strength, N
1.	1% $CaCO_3$	825	1300	10	2607
	0.5% alginate			20	2679
	11% water		1250	10	1764
				20	2248
			1150	10	627
				20	2064
			1050	10	350
				20	412
2.	1% $CaCO_3$	897	1300	10	1889
	0.5% alginate			20	2718
	10% water		1250	10	1777
				20	2515
			1150	10	660
				20	1866
			1050	10	336
				20	457
3.	1% $CaCO_3$	618	1300	10	4900
	0.25% HEC			20	5896
	10.5% water		1250	10	1820
				20	5619
			1150	10	336
				20	2604
			1050	10	163
				20	487
4.	2% $CaCO_3$	278	1300	10	4100
	0.25% MC			20	7971
	10% water		1250	10	1519
	no vacuum			20	4361
			1150	10	287
				20	1029
			1050	10	59
				20	190
5.	2% $CaCO_3$	390	1300	10	5276
	0.25% MC			20	6199
	10% water		1250	10	2352
				20	4867
			1150	10	421
				20	1369
			1050	10	85
				20	480

6.	1% $CaCO_3$	1328	1300	10	2564
	0.5% Na CMC			20	2854
	12% water		1250	10	1522
				20	1960
			1150	10	575
				20	
			1050	10	284
				20	438
7.	2% $CaCO_3$	1281	1300	10	3332
	0.25% CMC-7H			20	6697
	12% water		1250	10	3638
				20	4765
			1150	10	1235
				20	2350
			1050	10	337
				20	1464

+oven dry at 125°C